



## DECLARATION

I, Hisao SHIOZAWA, Patent Attorney, of SIKs & Co., 8th Floor, Kyobashi-Nishshoku Building, 8-7, Kyobashi 1-chome, Chuo-ku, Tokyo 104-0031 JAPAN hereby declare that I am the translator of the verified official copy of the documents in respect of an application for a patent filed in Japan on October 14, 1999 under Patent Application No.293003/1999 and that the following is a true and correct translation to the best of my knowledge and belief.

Date : October 9, 2002

Hisao SHIOZAWA

PATENT OFFICE

JAPANESE GOVERNMENT

This is to certify that the annexed is a true copy of the following application as filed with this office.

Date of Application : October 14, 1999

Application Number : Patent Application No. (Hei) 11-293003

Applicant(s) : HOYA CORPORATION

June 29, 2000

Takahiko KONDOH

Commissioner, Patent Office

Certification No. 2000-3053925

[Title] Application for Patent

[Reference Number] 99624H

[Filing Date] October 14, 1999

[Address] To the Commissioner of the Patent Office

[International Patent Classification] C03C 10/00

[Inventor]  
[Domicile or Residence]  
c/o HOYA CORPORATION  
7-5, Naka-Ochiai 2-chome, Shinjuku-ku, Tokyo

[Name] Xuelu ZOU

[Applicant for Patent]  
[Identification Number] 000113263  
[Name] HOYA CORPORATION

[Administrator]  
[Identification Number] 100092635  
[Patent Attorney]  
[Name] Hisao SHIOZAWA

[Subadministrator]  
[Identification Number] 100096219  
[Patent Attorney]  
[Name] Masazumi IMAMURA

[Priority Claimed on Prior Application]  
[Application Number] Patent Application No. (Hei) 11-193574  
[Filing Date] July 7, 1999

[Fee]  
[Number of register of payment] 007663  
[Amount] 21000

[List of Attached Documents]  
[Document's Name] Specification 1  
[Document's Name] Abstract 1  
[Number of General Power of Attorney] 9803325

[Proof] Required

[Document] Specification

[Title of the Invention] Substrate for information recording medium and magnetic recording medium composed of crystallized glass

5 [Claims]

[Claim 1] A crystallized glass substrate for information recording medium, characterized by comprising

SiO<sub>2</sub> : 35 — 65 mol%

Al<sub>2</sub>O<sub>3</sub> : 5 — 25 mol%

10 MgO : 10 — 40 mol%

TiO<sub>2</sub> : 5 — 15 mol%,

wherein the sum of the above components is at least equal to or more than 93 mol%, and by having a composition wherein a molar ratio (Al<sub>2</sub>O<sub>3</sub>/MgO) is less than 0.5, and characterized in that a main crystal phase is enstatite and/or its solid solution.

[Claim 2] The substrate according to claim 1, characterized in that the molar ratio (Al<sub>2</sub>O<sub>3</sub>/MgO) is equal to or more than 0.2.

20 [Claim 3] The substrate according to claim 1 or 2, characterized by comprising

SiO<sub>2</sub> : 40 — 60 mol%

Al<sub>2</sub>O<sub>3</sub> : 7 — 22 mol%

MgO : 12 — 35 mol%

25 TiO<sub>2</sub> : 5.5 — 14 mol%.

[Claim 4] The substrate according to any of claims 1 to 3, further comprising Y<sub>2</sub>O<sub>3</sub> in an amount equal to or less than 10 mol%.

30 [Claim 5] The substrate according to claim 4, comprising Y<sub>2</sub>O<sub>3</sub> in an amount ranging from 0.3 to 8 mol%.

[Claim 6] The substrate according to any of claims 1 to 5, further comprising ZrO<sub>2</sub> in an amount equal to or less than 6 mol%.

35 [Claim 7] The substrate according to any of claims 1 to 6, wherein quarts solid solution is not substantially

comprised as the main crystal phase.

[Claim 8] The substrate according to any of claims 1 to 7, wherein spinel is not substantially comprised as the crystal phase.

5 [Claim 9] The substrate according to any of claims 1 to 8, wherein the average particle diameter of the crystal particle is equal to or less than 0.5  $\mu\text{m}$ .

[Claim 10] The substrate according to any of claims 1 to 9, having a polished surface with a surface roughness Ra 10 equal to or less than 10  $\text{\AA}$ .

[Claim 11] The substrate according to any of claims 1 to 10, having a Young's modulus equal to or more than 140 Gpa.

[Claim 12] The substrate according to any of claims 1 to 11, further comprising an alkali metal oxide in an amount 15 equal to or less than 5 mol% and/or an alkali earth metal oxide in an amount equal to or less than 5 mol%.

[Claim 13] The substrate according to any of claims 1 to 11, further comprising 0.1 to 5 mol% of  $\text{K}_2\text{O}$  and/or 0.1 to 5 mol% of  $\text{SrO}$ .

20 [Claim 14] A crystallized glass substrate for information recording medium, characterized by being composed of crystallized glass comprising enstatite and/or its solid solution as a main crystal phase, and by having a polished surface with a surface roughness Ra equal to or less than 25 10  $\text{\AA}$ .

[Claim 15] The substrate according to claim 14, wherein the average particle diameter of the crystal particle is equal to or less than 0.5  $\mu\text{m}$ .

30 [Claim 16] The substrate according to claim 14 or 15, having a Young's modulus equal to or more than 140 Gpa.

[Claim 17] The substrate according to any of claims 14 to 16, wherein quarts solid solution is not substantially comprised as the main crystal phase.

35 [Claim 18] The substrate according to any of claims 14 to 18, wherein spinel is not substantially comprised as the

crystal phase.

[Claim 19] The substrate according to any of claims 1 to 18, wherein the information recording medium is a magnetic disk.

5 [Claim 20] An information recording medium comprising the substrate according to any one of claims 1 to 19 and a recording layer formed on the substrate.

[Claim 21] The information recording medium according to claim 20, wherein the recording layer is a magnetic recording 10 layer.

[Detailed description of the Invention]

[0001]

[Field of the Invention]

This invention relates to crystallized glasses 15 suitable for substrates which are used for information recording media, such as magnetic disks, optical disks and optical magnetic disks, substrates for information recording media composed of this crystallized glass, and information recording media using said substrate for 20 information recording medium.

[0002]

[Prior Art]

Major components of magnetic storage devices of computers and the like are a magnetic recording medium and 25 a magnetic head for magnetically recording and reproducing. Flexible disks and hard disks have been known as magnetic recording media. As substrates for hard disks, aluminum alloy has been mainly used. Recently, flying height of magnetic heads is markedly reduced as hard disk drives for 30 notebook personal computers are made smaller and their magnetic recording density made higher. Accordingly, extremely high precision has been demanded for the surface smoothness of magnetic disk substrates. However, in the case of aluminum alloy, it is difficult to produce smooth 35 surface more than a certain level of precision with an

aluminum alloy, because even though it is polished using highly precise abrasives and processing apparatuses, the polished surface may suffer from plastic deformation because of the low hardness. Even if the aluminum alloy 5 is plated with nickel-phosphorous, the surface roughness Ra cannot be made equal to or less than 5 Å. In addition, as hard disk drives are made smaller and thinner, a further smaller thickness of substrates for magnetic disks is also strongly desired. However, it is difficult to produce such 10 a thin disk with an aluminum alloy having a certain strength defined by specification of hard disk drives because of low strength and stiffness of aluminum alloy.

[0003]

Therefore, glass substrates for magnetic disks of 15 which high strength, high stiffness, high impact resistance and high surface smoothness are required have been developed. Among these, chemically reinforced glass substrates whose surfaces are strengthened by the ion exchange technique, crystallized glass substrates subjected to crystallization 20 treatment and the like have been known well.

As a chemically reinforced glass substrate by ion-exchange, for example, a glass disclosed in Japanese Patent Unexamined Publication No. Hei. 1-239036 has been known. This glass substrate reinforced by ion-exchange is 25 such a glass substrate for magnetic disks as the glass containing, indicated in terms of % by weight, 50-65 % of SiO<sub>2</sub>, 0.5-14 % of Al<sub>2</sub>O<sub>3</sub>, 10-32 % of R<sub>2</sub>O where R is an alkali metal ion, 1-15 % of ZnO and 1.1-14 % of B<sub>2</sub>O<sub>3</sub> is reinforced by forming a crushing stress layer on the glass substrate 30 surface with an ion exchange method by an alkali ion.

[0004]

In addition, as a crystallized glass, for example, a glass disclosed in Japanese Patent No. 2516553 is exemplified. This crystallized glass is such a 35 crystallized glass for magnetic disks which contains,

indicated in terms of % by weight, 65-83 % of SiO<sub>2</sub>, 8-13 % by Li<sub>2</sub>O, 0-7 % of K<sub>2</sub>O, 0.5-5.5 % of MgO, 0-5 % of ZnO, 0-5 % of PbO (provided that MgO + ZnO + PbO is 0.5-5 %), 1-4 % of P<sub>2</sub>O<sub>5</sub>, 0-7 % of Al<sub>2</sub>O<sub>3</sub> and 0-2 % of As<sub>2</sub>O<sub>3</sub> + Sb<sub>2</sub>O<sub>3</sub>, and contains 5 micro crystalline particles of Li<sub>2</sub>O · 2SiO<sub>2</sub> as main crystals.

Moreover, a crystallized glass is also disclosed in Japanese Patent Unexamined Publication No. Hei. 7-291660. This crystallized glass is obtained by heat treatment after fusion and forming a glass which is composed of, indicated 10 in terms of % by weight, 38-50 % of SiO<sub>2</sub>, 18-30 % of Al<sub>2</sub>O<sub>3</sub>, 10-20 % of MgO, provided that having a composition containing, indicated in terms of weight ratio, 1.2-2.3 of Al<sub>2</sub>O<sub>3</sub>/MgO, 0%-5% of B<sub>2</sub>O<sub>3</sub>, 0%-5% of CaO, 0%-5% of BaO, 0%-5% of SrO, 0.5%-7.5% of ZnO, 4%-15% of TiO<sub>2</sub>, 0%-5% of ZrO<sub>2</sub> and 0%-2% 15 of As<sub>2</sub>O<sub>3</sub> and/or Sb<sub>2</sub>O<sub>3</sub>. This glass is a cordierite based crystallized glass characterized by containing cordierite based crystals as main crystals. Moreover, a substrate for magnetic disks composed of this crystallized glass is also disclosed.

20 [0005]

In addition, a crystallized glass is also disclosed in Japanese Patent Unexamined Publication No. Hei. 9-77531 (U.S. Patent No. 5,476,821). This crystallized glass is a glass ceramic product having the Young's modulus in the 25 range of about  $14 \times 10^6$  to about  $24 \times 10^6$  psi (96 to 165 GPa) and the fracture toughness of more than 10 MPa·m<sup>1/2</sup>. In addition, this crystallized glass is composed of crystalline phase laminated body which is mainly composed of crystals having a spinel structure and a uniform size 30 and dispersing uniformly in a siliceous-rich residual glass matrix. This is a glass ceramic which is substantially composed of, indicated in terms of % by weight using oxides as a standard, 35-60% of SiO<sub>2</sub>, 20-35 % of Al<sub>2</sub>O<sub>3</sub>, 0-25% of MgO, 0-25% of ZnO, 0-20% of TiO<sub>2</sub>, 0-10% of ZrO<sub>2</sub>, 0-2% of 35 Li<sub>2</sub>O and 0-8% of NiO. This has at least about 10% of a total

amount of MgO+ZnO and may contain equal to or less than 5% of an optional component selected from the group consisting of BaO, CaO, PbO, SrO, P<sub>2</sub>O<sub>5</sub>, B<sub>2</sub>O<sub>3</sub> and Ga<sub>2</sub>O<sub>3</sub>, in the range of 0 to 5 % of R<sub>2</sub>O selected from the group consisting of Na<sub>2</sub>O, K<sub>2</sub>O, Rb<sub>2</sub>O and Cs<sub>2</sub>O, and in the range of 0 to 8 % of transition metals. In the case of containing less than about 25 % of Al<sub>2</sub>O<sub>3</sub>, this is a glass ceramic having a composition in which the total amount of TiO<sub>2</sub>+ZrO<sub>2</sub>+NiO is equal to or more than 5 %. The substrate for magnetic disks composed of this glass ceramic is disclosed.

[0006]

In addition, a crystallized glass is also disclosed in U.S. Patent No. 5,491,116. This crystallized glass is a glass ceramic product having the fracture coefficient of at least about 15,000 psi, the Knoop hardness exceeding about 760 KHN, the Young's modulus of more than about 20 × 10<sup>6</sup> psi and the fracture toughness of more than 1.0 MPa·m<sup>1/2</sup>. The main crystal phase is composed of enstatite or its solid solution and spinel (spinel structure crystal), and the crystallized glass contains at least 92% of the composition substantially consisting of, indicated in terms of % by weight, 35-60% of SiO<sub>2</sub>, 10-30% of Al<sub>2</sub>O<sub>3</sub>, 12-30% of MgO, 0-10% of ZnO, 5-20% of TiO<sub>2</sub> and 0-8% of NiO. Moreover, the substrate for magnetic disks composed of this crystallized glass is also disclosed. It is to be noted that the same glass as the crystallized glass disclosed in aforementioned patent is also disclosed in Journal of Non-Crystalline Solids 219(1997) 219-227.

In addition, those in which a main crystal comprises enstatite as a main crystal phase is disclosed in Japanese Patent No. 2,648,673, but it is not disclosed that those are employed for magnetic disks. If this crystallized glass is a glass ceramic product employed at a temperature equal to or higher than 1200 °C and comprising crystal in an amount at least 90 volume %, this product comprises enstatite as

a main crystal phase, the second crystal phase composed of at least one selected from  $\beta$ -spodumene solid solution, Ba-osumilite, celsian, anorthite, cristobalite, and strontium feldspar, which has a composition substantially consisting of, indicated in terms of % by weight using oxides as a standard, 20-35% of MgO, 2-12% of Al<sub>2</sub>O<sub>3</sub>, 40-70% of SiO<sub>2</sub>, as well as at least one metal oxide in the prescribed amount selected from the group consisting of 0-2% of Li<sub>2</sub>O, 0-4% of CaO, 0-12% of SrO and 0-17% of BaO, wherein if Li<sub>2</sub>O exists alone, at least 0.5% is required, and if Li<sub>2</sub>O does not exist, at least 1% of CaO and/or SrO and/or BaO is required, and the composition does not substantially Na<sub>2</sub>O, K<sub>2</sub>O and fluoride.

[0007]

15 [Problems to be solved by the Invention]

However, recently, along with making hard disks smaller and thinner and making recording density higher, it is rapidly developed to make flight height of magnetic heads smaller and revolution speed of disks higher. Thereby, substrate materials are more strictly required the strength, the Young's modulus, the smoothness of the surface and the like. In particular, by making the information recording density of 3.5-inch hard disks for personal computers and servers higher, the surface smoothness and the surface flatness of the substrate materials are strictly required. In addition, corresponding to the higher data processing speed, it is required to set the winding number of the disks equal to or higher than 10,000 rpm. Thus, the requirement for stiffness of substrate materials becomes increasingly severer, and the limitation of conventional aluminum substrates already becomes obvious. In future, as long as it is necessarily demanded to make the capacity of hard disks higher and to make the revolution speed of hard disks higher, it is clear that the substrate materials for magnetic recording medium is strongly required to exhibit higher

Young's modulus, higher strength, more excellent surface flatness, higher impact resistance and the like.

[0008]

Demands for high Young's modulus can be explained on the basis of the following fact. That is, with recent smaller size, higher capacity and higher speed of HDDs, it is expected that the thickness of 3.5-inch disks currently used of 0.8 mm will be made smaller to 0.635 mm, and 0.635 mm of current 2.5-inch disks to 0.43 mm, or even to 0.38 mm. Revolution speed of substrates is also expected to be made faster from the current maximum speed of 10,000 rpm to 14,000 rpm. As substrates for such magnetic recording media become thinner, they become more likely to suffer deflexion, undulation and warp, and it is expected that, as the revolution speed becomes higher, stress loaded on the substrates (force exerted by wind pressure caused by rotation of disks) will become larger. Based on the theory of dynamics, the deflexion  $W$  of a disk receiving load of  $P$  per unit area is represented by the following formula:

[0009]

[Number 1]

$$W \propto \frac{Pa^4}{h^3E}$$

[0010]

However,  $a$  represents an outer diameter of disk,  $h$  represents a thickness of substrate and  $E$  represents Young's modulus of disk material.

In static state, force loaded on the disk is the gravitation alone is, and the deflexion  $W$  is represented, if  $d$  represents a specific gravity of disk material, by the following formula:

[0011]

[Number 2]

$$W \propto \frac{hda^4}{h^3E} = \frac{da^4}{h^2E} = \frac{a^4}{h^2G}$$

[0012]

In the above, G is a specific elastic modulus of disk material (= Young's modulus/specific gravity).

On the other hand, supposing that the gravitational force component is balanced by centrifugal force component and can be ignored in rotating state of disk, force loaded on the disk may be considered only wind pressure caused by the rotation of the disk. The wind pressure is represented as a function of disk revolution speed and said to be proportional to the second power of the speed. Accordingly, the deflexion W when the disk is rotating at high speed is represented by the following formula:

[0013]

[Number 3]

$$w \propto \frac{(rpm)^2 a^4}{h^3E}$$

[0014]

From these results, it is proven that, in order to suppress the deflexion W of substrate to be rotated at a high speed, a material of high Young's modulus E is required. According to the present inventors' calculation, when the thickness of 2.5-inch substrate is made smaller from 0.635 mm to 0.43 mm, and the thickness of 3.5-inch substrate from 0.8 mm to 0.635 mm, it is required that the specific elastic modulus of substrate materials is at least equal to or higher than 37 MNm/kg. In addition, when the current revolution speed of 3.5-inch high-end substrates of 7200 rpm is made faster to prospective 10000 rpm, an aluminum substrate having Young's modulus of around 70 GPa cannot meet such a high speed, and new substrate materials having the Young's modulus at least equal to or higher than 110 GPa are required.

As the specific elastic modulus or Young's modulus of substrate material becomes higher, not only stiffness of substrates becomes higher, but also impact resistance and strength of substrates become higher. Therefore, a glass 5 material having high specific elastic modulus and high Young's modulus is strongly desired in the field of HDD production.

[0015]

However, such a chemically reinforced glass as 10 disclosed in Japanese Patent Unexamined Publication No. Hei.1-239036 mentioned above has the Young's modulus of about 80 Gpa, therefore, it is obvious that it cannot meet the strict demand for hard disks in future. As for conventional chemically reinforced substrate glasses, 15 alkali ions are introduced in a large amount in the glass for ion exchange, so that the reinforced glasses mostly have the low Young's modulus (90 Gpa). Moreover, due to also having low stiffness, it cannot meet 3.5-inch high-end disk substrates and thinner disk substrates. In addition, 20 the glass chemically reinforced by ion exchange contains large amount of alkali components. Thus, if it is used for long hours under the circumstance of high temperature and high humidity, alkali ions deposit from parts including thin magnetic films or exposing glasses, such as a pinhole 25 part of a magnetic film or a circumference of a magnetic film. It has a disadvantage that this triggers a corrosion or decomposition of the magnetic films. In the producing process of the magnetic recording medium, after providing a magnetic layer on the glass substrate, certain heat 30 treatment may be carried out in order to improve characteristics such as coercive force of the magnetic layer. However, the conventional ion-exchanged reinforced glass mentioned above has at most 500 °C of the glass transition 35 temperature, so that it has poor heat resistance. Thereby, it has a problem that higher coercive force cannot be

obtained.

[0016]

In addition, the conventional crystallized glass as disclosed in Japanese Patent Publication No.2516553 mentioned above is superior a little in the Young's modulus and heat resistance than aforementioned chemically reinforced glass substrate. However, the surface roughness is equal to or higher than 10 Å, thereby the surface smoothness is poor, so that it is limited to make the flying height smaller. Therefore, it has a problem that it cannot meet higher magnetic recording density. Moreover, the Young's modulus is about 90 to 100 Gpa at most, so that it also cannot meet 3.5-inch high-end disk substrates and thinner disk substrates.

In addition, the crystallized glass disclosed in Japanese Patent Unexamined Publication No.Hei.7-291660 mentioned above has the Young's modulus of about 100 to 130 Gpa at most, therefore, it cannot be said that it is sufficient. Moreover, it has only such a surface smoothness as having the Young's modulus of about 8 Å, resulting in poor smoothness. Additionally, the temperature of glass liquid phase is high which is 1400 °C, so that it has a disadvantage of difficulty in producing.

[0017]

Moreover, the crystallized glass disclosed in Japanese Patent Unexamined Publication No.Hei.9-77531 mentioned above has a disadvantage of large difficulty in polishing because the main crystals are spinel. Additionally, the temperature of glass liquid phase is high which is 1400 °C or more, so that it has a disadvantage of difficulty in melting and forming at high temperature.

Moreover, a large amount of enstatite is contained together with spinel in the crystallized glass disclosed in U.S. Patent No.5,491,116 and Journal of Non-Crystalline Solids 219(1997) 219-227. Accordingly, it can be

considered that easiness in polishing is more improved than the crystallized glass disclosed in Japanese Patent Unexamined Publication No. Hei.9-77531. However, because spinel is still contained therein, it is difficult to say 5 that it has sufficient. That is, it still takes a long time for polishing required for obtaining desirable surface roughness, so that it has a problem of inferior productivity.

[0018]

Moreover, because a glass disclosed in Japanese Patent 10 No. 2648673 is a fire-resistant glass ceramic for the purpose of using temperature equal to or higher than 1200 °C, it is difficult to be used as a substrate for information recording medium. That is, it is difficult to be produced due to high melting temperature, moreover, the surface 15 smoothness required for information recording medium cannot be obtained due to large crystal size.

[0019]

Therefore, it is an object of the present invention to provide a crystallized glass suitable for a substrate 20 for information recording medium, such as magnetic disks and the like, which has higher Young's modulus, higher strength and higher fire-resistance, superior in the surface smoothness, surface homogeneity and surface processability with considering demands that a substrate 25 for magnetic recording medium will be made thinner and will have high strength, high heat resistance, high impact resistance and the like in near future, as well as which can be cheaply produced due to comparatively low temperature of the glass liquid phase.

Moreover, it is also an object of the present invention 30 to provide a substrate for information recording medium, such as magnetic disks and the like, composed of aforementioned crystallized glass, which has higher Young's modulus, higher strength and higher fire-resistance, 35 superior in the surface smoothness, surface homogeneity

and surface processability, as well as which can be cheaply produced due to comparatively low temperature of the glass liquid phase, and information recording media using the substrate, such as magnetic disks.

5 [0020]

[Means for solving problems]

To solve objects mentioned above, the inventors carried out various examinations, as results, it is found that crystallized glass suitable for substrate for 10 information recording medium which has high Young's modulus equal to or higher than 140 GPa, good surface smoothness, excellent surface processability, and comparatively low liquid temperature with a composition in which a molar ratio (Al<sub>2</sub>O<sub>3</sub>/MgO) is less than 0.5 can be obtained by employing 15 a TiO<sub>2</sub> component as a nucleus-forming agent and making a molar ratio (Al<sub>2</sub>O<sub>3</sub>/MgO) less than 0.5 in a SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-MgO based glass in which a main crystal phase is enstatite and/or its solid solution. Then, they accomplished the present invention.

20 The present invention relates to a crystallized glass substrate for information recording medium characterized by comprising

SiO<sub>2</sub> : 35 — 65 mol%

Al<sub>2</sub>O<sub>3</sub> : 5 — 25 mol%

25 MgO : 10 — 40 mol%

TiO<sub>2</sub> : 5 — 15 mol%,

wherein the sum of the above components is at least equal to or more than 93 mol%, and by having a composition wherein a molar ratio (Al<sub>2</sub>O<sub>3</sub>/MgO) is less than 0.5, and characterized 30 in that a main crystal phase is enstatite and/or its solid solution.

The present invention further relates to a crystallized glass substrate for information recording medium, characterized by consisting of crystallized glass 35 comprising enstatite and/or its solid solution as a main

crystal phase, and by having a polished surface with a surface roughness Ra equal to or less than 10 Å.

Further, the present invention relates to an information recording medium comprising a recording layer 5 formed on the substrate composed of the crystallized glass according to the present invention.

[0021]

[Crystallized glass]

Each of the components in the crystallized glass 10 constituting the glass of the present invention will be explained in the followings.

[0022]

$\text{SiO}_2$  is a component of the network structure of glass, in addition, it is also a component of enstatite having 15 a composition of  $\text{MgO} \cdot \text{SiO}_2$ , and enstatite solid solution having a composition of  $(\text{Mg} \cdot \text{Al})\text{SiO}_3$ , which are the main deposited crystals. Because the melted glass is very unstable in the case of having the content of  $\text{SiO}_2$  of less than 35 %, it is afraid that it cannot be molded at high 20 temperature, in addition, crystals as mentioned above become difficult to deposit. If the content of  $\text{SiO}_2$  is less than 35 %, the chemical durability and heat resistance of residual glass matrix phase tend to deteriorate. On the other hand, if the content of  $\text{SiO}_2$  exceeds 65 %, enstatite 25 becomes difficult to deposit as main crystals and the Young's modulus of glass tends to be lowered rapidly. Therefore, the content of  $\text{SiO}_2$  ranges from 35 to 65 % in view of kinds of the deposited crystals, the deposited amount thereof, chemical durability, heat resistance, molding 30 characteristics and productivity. From the viewpoint that such a crystallized glass can be obtained as having more preferable physical characteristics, the content of  $\text{SiO}_2$  preferably ranges from 40 to 60 %.

[0023]

35  $\text{Al}_2\text{O}_3$  is a medium oxide of glass and contributes to

improvement of glass surface hardness. However, if the glass content is less than 5 %, chemical durability of glass matrix phase is lowered and the strength required to substrate materials tends to be difficult to be obtained.  
5 On the other hand, if the content of  $\text{Al}_2\text{O}_3$  exceeds 25 %, enstatite as a main crystal phase becomes difficult to deposit as well as the glass become difficult to melt due to the high melting temperature. In addition, the glass has a tendency to be easily devitrified and to increase  
10 molding difficulty. Accordingly, it is appropriate that the content of  $\text{Al}_2\text{O}_3$  ranges from 5 to 25%, preferably from 7 to 22% in view of melting characteristics, molding characteristics at a high temperature, kinds of deposited crystals of glass and the like.

15 [0024]

$\text{MgO}$  is a glass modifying component, in addition, a main component of crystals of enstatite having a composition of  $\text{MgO} \cdot \text{SiO}_2$  and its solid solution. If the content of  $\text{MgO}$  is less than 10 %, crystals as mentioned above are difficult  
20 to deposit, having a high devitrifying tendency and a high melting temperature of the glass, as well as the working temperature width of glass molding tends to be narrower. On the other hand, if the content of  $\text{MgO}$  exceeds 40 %,  
25 viscosity of glass at a high temperature is rapidly lowered, thereby, it becomes thermally unstable, productivity deteriorates and the Young's modulus and durability tend to be lowered. Then, it is appropriate that the content of  $\text{MgO}$  ranges from 10 to 40%, preferably from 12 to 35% in view of productivity, chemical durability, high  
30 temperature viscosity and strength of glass.

[0025]

Provided that the contents of  $\text{MgO}$  and  $\text{Al}_2\text{O}_3$  are adjusted so that the molar ratio ( $\text{Al}_2\text{O}_3/\text{MgO}$ ) is less than 0.5. Because the Young's modulus of the crystallized glass rapidly lowers  
35 if the molar ratio ( $\text{Al}_2\text{O}_3/\text{MgO}$ ) becomes equal to or higher

than 0.5. By adjusting the  $\text{Al}_2\text{O}_3/\text{MgO}$  less than 0.5, such a crystallized glass can be also obtained as having high Young's modulus equal to or higher than 150 GPa.  $\text{Al}_2\text{O}_3/\text{MgO}$  is preferably less than 0.45. However, if the molar ratio of  $\text{Al}_2\text{O}_3/\text{MgO}$  is adjusted to excessively low, it is afraid that viscosity of glass at a high temperature tends to be lowered and the crystal particle becomes large. Therefore, it is appropriate that  $\text{Al}_2\text{O}_3/\text{MgO}$  ratio is equal to or higher than 0.2, preferably equal to or higher than 0.25.

10 [0026]

$\text{TiO}_2$  is a nucleation agent for deposition of crystalline phase of enstatite having a composition of  $\text{MgO} \cdot \text{SiO}_2$ , and enstatite solid solution having a composition of  $(\text{Mg} \cdot \text{Al})\text{SiO}_3$ . Moreover, if the content of  $\text{SiO}_2$  is small, 15  $\text{TiO}_2$  also has an effect of suppressing glass devitrification. However, if the content of  $\text{TiO}_2$  is less than 5 %, the effect as the nucleation agent for the main crystals cannot be obtained sufficiently, thereby, crystallization causes at the surface of the glass and it tends to be difficult to 20 produce homogenous crystallized glass. On the other hand, if the content of  $\text{TiO}_2$  exceeds 15 %, phase separating and devitrification of the glass occur due to excessively low glass viscosity at a high temperature, so that glass productivity tends to extremely deteriorate. Therefore, 25 it is appropriate that the content of  $\text{TiO}_2$  ranges from 5 to 15 %, preferably from 5.5 to 14 %.

[0027]

In the crystallized glass of the present invention,  $\text{Y}_2\text{O}_3$  is not an essential component. However, as seen in 30 the following examples, the Young's modulus of the crystallized glass can be increased to about 10 GPa as well as the temperature of liquid phase can be decreased to about from 50 to 100 °C by introducing, for example, 2 % of  $\text{Y}_2\text{O}_3$ . That is, characteristics and productivity of glass can be 35 improved remarkably by introducing a little amount of  $\text{Y}_2\text{O}_3$ .

If the content of  $\text{Y}_2\text{O}_3$  is equal to or higher than 0.3 %, the effect of  $\text{Y}_2\text{O}_3$  can be obtained. The content of  $\text{Y}_2\text{O}_3$  is preferably equal to or higher than 0.5 %. However,  $\text{Y}_2\text{O}_3$  has an effect of suppressing the growth of the main crystal contained in the aforementioned glass. Therefore, if the content of  $\text{Y}_2\text{O}_3$  is excessively high, the surface is easily crystallized in the heat-treatment carried out for the purpose of the glass crystallization, so that it tends not to produce a desired crystallized glass. From such viewpoints, it is appropriate that the content of  $\text{Y}_2\text{O}_3$  is equal to or less than 10 %. In particular, the content of  $\text{Y}_2\text{O}_3$  is preferably equal to or less than 8 %, more preferably equal to or less than 3 %.

[0028]

Moreover, the crystallized glass of the present invention can contain  $\text{ZrO}_2$ .  $\text{ZrO}_2$  can play a great role to improvement of glass stability, especially in a glass containing a large amount of  $\text{MgO}$ . In addition, it also works as a nucleation agent and it contributes, as a helper of  $\text{TiO}_2$ , to make the crystal particles fine with promoting phase separation of glass during pretreatment. However, if the content of  $\text{ZrO}_2$  exceeds 6 %, it is afraid that melting characteristics at a high temperature and homogeneity of glass deteriorate, so that the amount to be introduced is equal to or less than 6 %. Moreover, the content of  $\text{ZrO}_2$  is preferably equal to or less than 5% in view of melting characteristics of glass at a high temperature and homogeneity of the crystal particles.

[0029]

In the crystallized glass of the present invention, the sum of  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{MgO}$  and  $\text{TiO}_2$  is equal to or higher than 93 % from the viewpoint of characteristics such as the high Young's modulus and keeping homogenous crystalline characteristics. The sum of  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{MgO}$  and  $\text{TiO}_2$  is preferably equal to or higher than 95 %.

[0030]

If within above range, as components other than said components, it may contain such components as alkali metal oxides (for example, Li<sub>2</sub>O, Na<sub>2</sub>O, K<sub>2</sub>O or the like) and/or alkali-earth metal oxides (for example, CaO, SrO, BaO or the like) in the range of not deteriorating desirable characteristics of the crystallized glass. Alkali metal oxides and/or alkali-earth metal oxides can be produced from nitrates as glass raw materials. If Sb<sub>2</sub>O<sub>3</sub> is used as a degassing agent in glass producing, Pt is easily included into glass from a crucible made of Pt for glass melting. By using nitrates as glass raw materials, inclusion of Pt into glass can be suppressed. It is preferable that the content of alkali metal oxides and alkali-earth metal oxides is respectively equal to or higher than 0.1 mol% from the viewpoint of obtaining said effect. However, if containing alkali metal oxides, its content is appropriately equal to or less than 5 mol% because alkali metal oxides tend to lower the Young's modulus. In addition, in the case of containing alkali-earth metal oxides, the content of the alkali-earth metal oxides is appropriately equal to or less than 5 mol% because of their tendency to increase crystal particles. In the case of containing alkali metal oxides, especially, the content of K<sub>2</sub>O preferably ranges from 0.1 to 5 mol%, preferably from 0.1 to 2 mol%, more preferably from 0.1 to 1 mol%. In the case of containing alkali-earth metal oxides, especially, the content of SrO ranges from 0.1 to 5 mol%, preferably from 0.1 to 2 mol%.

[0031]

In addition, As<sub>2</sub>O<sub>3</sub> and/or Sb<sub>2</sub>O<sub>3</sub> can be contained as a degassing agent for attempting glass homogenization. According to viscosity at a high temperature changing by the composition of glass, more homogenous glass can be obtained by adding an adequate amount of As<sub>2</sub>O<sub>3</sub>, Sb<sub>2</sub>O<sub>3</sub> or As<sub>2</sub>O<sub>3</sub>+Sb<sub>2</sub>O<sub>3</sub> to glass. However, if excessively large amount

of the degassing agent is added, the Young's modulus tends to be lowered by increasing the specific gravity of glass. Moreover, it may react with a platinum crucible for melting, and thereby damaging the crucible. Therefore, it is 5 appropriate that the amount of the degassing agent added is equal to or less than 2 %, preferably equal to or less than 1.5 %.

Impurities in raw materials, such as Cl, F, SO<sub>3</sub> and the like which become a glass cleaning agent, can be contained 10 other than aforementioned basic components if the content is respectively equal to or less than 1 % where deterioration of the characteristics of the crystallized glass of the present invention is avoidable.

In addition, it is preferable that the crystallized 15 glass of the present invention does not contain ZnO and NiO. Because ZnO facilitates production of spinel, hard crystals. In addition, not containing NiO is desirable from the viewpoint that NiO facilitates production of spinel, as well as from the viewpoint that NiO is such a component 20 as affecting the environment.

[0032]

The main crystal phase in the crystallized glass of the present invention is, for example, enstatite (including enstatite solid solution) having a composition of MgO · 25 SiO<sub>2</sub> and (Mg · Al)SiO<sub>3</sub>. In addition, enstatite includes clino-enstatite, proto-enstatite and enstatite. Moreover, other than aforementioned crystals, titanates, mullite, forsterite, cordierite and the other crystals can be comprised. Enstatite has such characteristics as the 30 crystallized glass containing enstatite as main crystals can be polished very easily due to low hardness (where the Mohs' scale of hardness is 5.5), and it can obtain desirable surface roughness in relatively short time. Moreover, it can be considered that enstatite can provide a glass with 35 high Young's modulus even if the particle size is small

because glass components easily penetrate into its voids due to the crystal structure of chain or layer. In addition, spinel is not substantially comprised in the glass of the present invention. It is considered that because spinel  
5 is harder (where the Mohs' scale of hardness is 8) than enstatite, it deteriorates the effect of easy polishing. The glass of the present invention does not substantially contain quartz solid solution.

[0033]

10 It is to be noted that, in the present invention, the main crystal phase is an essential crystal phase for achieving the effect of the present invention, and contained in an amount equal to or higher than 50 volume% among crystals (the kind of which can be defined by X-ray diffraction)  
15 in the glass. It is to be noted that, in many cases in the present invention, enstatite and/or its solid solution is contained in an amount equal to or higher than 70 volume% in the crystals, in some cases, it may be contained in an amount equal to or higher than 80 volume%, further equal to or higher than 90 volume%. In addition, in the present  
20 invention, the ratio of crystals in glass ranges from about 20 to 70 %.

Moreover, the average value of size of crystals contained in the crystallized glass of the present invention  
25 (particle diameter) is preferably equal to or less than  $0.5 \mu\text{m}$ , more preferably equal to or less than  $0.3 \mu\text{m}$ , further preferably equal to or less than  $0.1 \mu\text{m}$ . The average value of size of crystals contained in the crystallized glass of the present invention is most preferably equal to or  
30 less than 50nm. If the average value of crystal size exceeds  $0.5 \mu\text{m}$ , not only lowering the mechanical strength of glass but also the loss of crystals during polishing process causes, thereby it is afraid that the surface roughness of glass deteriorates. Such a control of size of crystal particles  
35 can be done mainly by changing kinds of contained crystal

phase and heat treatment conditions mentioned below. In the present invention, it is possible to obtain such micro crystal size under the heat treatment conditions under which the main crystal phase of enstatite and/or its solid solution 5 which are essential components in the present invention can be obtained.

[0034]

[Production method for crystallized glass and substrate]

The crystallized glass and the substrate of the present 10 invention can be produced by publicly known production methods of glass. For example, glass materials of a given ratio can be melted by the high temperature melting method, i.e., melted in air or inert gas atmosphere, homogenized by bubbling, addition of degassing agent, stirring or the like and molded into plate glass by well-known press method, download molding or the like. Then, glass molding products 15 of a desired size and shape can be obtained by processing such as cutting and polishing.

[0035]

20 The glass molding product thus obtained is subjected to heat treatment method for crystallization. The heat treatment method is not especially limited and it can be selected properly according to the content of crystallization promoting agents, glass transition 25 temperature, crystallization peak temperature or the like. However, many crystalline nuclei are generated by heat treatment at relatively low temperature (for example, 700 to 850°C) in the initial stage. After that, it is preferable that the crystals are grown by elevating the temperature 30 to from 850 to 1150 °C from the viewpoint of obtaining micro crystals. During producing the crystallized glass of the present invention, heat treatment schedule or glass composition can be sequentially changed to control the size 35 of the deposited crystals and the amount of crystals, thereby, characteristics of the crystallized glass can be remarkably

adjusted. In addition, in the present invention, the admissible temperature range of heat treatment for producing crystalline nuclei and heat treatment for growth of crystals in which such a crystallized glass as having the same Young's modulus and the same crystal size or the same crystallization homogeneity can be produced is a temperature width equal to or higher than 30 °C, so that production process for crystallization can be controlled easily.

10 [0036]

Moreover, in the present invention, such a heat treatment conditions are adjusted to conditions under which enstatite having a composition of  $MgO \cdot SiO_2$  and enstatite solid solution having a composition of  $(Mg \cdot Al)SiO_3$  are deposited as main crystals. It is to be noted that other crystals such as forsterite, cordierite, titanates and mullite may be deposited other than these main crystals, but the conditions under which enstatite and its solid solution are deposited is adopted. As such conditions, the crystallization temperature is controlled as low as possible. For example, it is appropriately equal to or less than 1150 °C, preferably equal to or less than 1100 °C. In addition, the temperature of nuclei producing process appropriately ranges from 0 to 60 °C higher than the glass transition temperature, preferably from 10 to 50 °C higher than the glass transition temperature.

20 [0037]

The molding product of the crystallized glass after subjected to heat treatment may be polished, if necessary, 30 and polishing method is not especially limited. For example, it can be polished by publicly known methods using synthetic sharpening particle, such as synthetic diamond, silicon carbide, aluminum oxide and boron carbide, and natural sharpening particle, such as natural diamond and cerium 35 oxide. The substrate for information recording medium of

the present invention composed of the crystallized glass of the present invention can be obtained by making the molding product in a substrate shape in the aforementioned method.

5       The substrate composed of the crystallized glass of the present invention preferably has the surface smoothness as having the average roughness Ra equal to or less than 10 Å measured with AFM. Especially, in the case of using the crystallized glass of the present invention as a magnetic 10 disk substrate, the average roughness Ra of the surface significantly influences recording density of the magnetic disk. If the surface roughness exceeds 10 Å, high recording density is hardly obtained. The surface roughness of the substrate composed of the crystallized glass of the present 15 invention is more preferably equal to or less than 7 Å, further preferably equal to or less than 5 Å in view of obtaining high recording density of magnetic disks.

[0038]

20      The substrate composed of the crystallized glass of the present invention in which enstatite and its solid solution are contained as main crystals is useful as a magnetic disk substrate because it has high strength, high hardness and high Young's modulus as well as being excellent in chemical durability and heat resistance. Moreover, the 25 crystallized glass of the present invention is alkali free or low-alkali, so that if used as a magnetic disk substrate, corrosion of a magnetic film with the substrate can be greatly decreased, thereby the magnetic film can be kept in the best condition.

30 [0039]

35      The crystallized glass substrate of the present invention is composed of SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-MgO based glass in which the main crystals are enstatite and/or its solid solution, as mentioned above. Other than this, the present invention includes the crystallized glass substrates for information

recording medium composed of the crystallized glass which is a glass other than  $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-MgO}$  based glass, and in which the main crystals are enstatite and/or its solid solution, as well as having a polished plane with the surface roughness  $R_a$  equal to or less than  $10\text{\AA}$ . It is to be noted that the main crystals herein are such crystals as contained in an amount equal to or higher than 50 volume% among crystals (the kind of which can be defined by X-ray diffraction) in the glass. In many cases, enstatite and/or its solid solution is contained in an amount equal to or higher than 70 volume% in the crystals, in some cases, it may be contained in an amount equal to or higher than 80 volume%, further equal to or higher than 90 volume%. In addition, in the crystallized glass, the ratio of crystals in glass ranges from about 20 to 70 %.

[0040]

In the crystallized glass substrate in this embodiment, the average value of the size (particle diameter) of the crystals contained in the crystallized glass is preferably equal to or less than  $0.5\text{ }\mu\text{m}$ , more preferably equal to or less than  $0.3\text{ }\mu\text{m}$ , further preferably equal to or less than  $0.1\text{ }\mu\text{m}$ . The average value of the size of the crystals contained in the crystallized glass is the most preferably equal to or less than 50 nm. If the average value of the crystal size exceeds  $0.5\text{ }\mu\text{m}$ , it is afraid that not only lowering the mechanical strength of the glass but also the loss of crystals during polishing process causes, thereby the glass surface roughness deteriorates. Such a control of the size of crystal particles is carried out mainly by selecting kinds of the contained crystal phase and heat treatment condition mentioned below. However, in the present invention, it is possible to obtain the microcrystal size as mentioned above in the heat treatment conditions under which the main crystals of enstatite and/or its solid solution which are essential in the present invention are

obtained.

The crystallized glass substrate in this embodiment preferably has a Young's modulus equal to or higher than 140 GPa as the substrate use for rotation at a high speed.  
5 In addition, it is preferable that the crystallized glass substrate of the present invention substantially does not contain quartz solid solution as main crystals, and substantially does not contain spinel as a crystal phase. Because spinel is hard crystals compared with enstatite  
10 (having the Mohs' scale of hardness of 8), it is difficult to be polished and the polished plane having a surface roughness Ra equal to or less than 10 Å is hardly obtained.  
[0041]

The magnetic disk substrate composed of the crystallized glass of the present invention can satisfy all of surface smoothness, flatness, strength, hardness, chemical durability, heat resistance and the like required as a magnetic disk substrate. In addition, the Young's modulus is about twice larger than the conventional  
20 crystallized glass ( $\text{Li}_2\text{O}\text{-SiO}_2$  based crystallized glass), so that it is possible to suppress the deflexion caused by high revolution speed of the disk low, thereby it is suitable as a substrate material to achieve high TPI hard disks.

Because the crystallized glass of the present invention is excellent in heat resistance, surface smoothness, chemical durability, optical characteristics and mechanical strength, it can be used suitable as substrates for information recording medium such as  
30 magnetic disks, glass substrates for optical magnetic disks and electron optical glass substrates such as optical disks.  
[0042]

#### [Explanation of magnetic disk]

The information recording medium of the present invention is characterized by having the substrate of the  
35

present invention and a recording layer formed on said substrate. A magnetic disk (hard disk) in which at least a magnetic layer is formed on the main surface of the substrate composed of the crystallized glass of the present 5 invention will be explained below.

As layers other than the magnetic layer, underlying layer, protective layer, lubricating layer, unevenness control layer and the like are exemplified in view of functions, and optionally formed if necessary. Each of 10 these layers can be formed by various thin film-forming techniques. Material for the magnetic layer is not particularly limited. For example, as a magnetic layer, in addition to Co-based one, ferrite-based one, iron-rare 15 earth metal-based one and the like can be exemplified. The magnetic layer may be either for horizontal magnetic recording or vertical magnetic recording.

Specific examples of the magnetic layer include, for example, magnetic thin films containing Co as a main component such as CoPt, CoCr, CoNi, CoNiCr, CoCrTa and CoPtCr, 20 and CoNiCrPt, CoNiCrTa, CoCrPtTa, CoCrPtSio and the like. The magnetic layer may have a multi-layered structure in which the magnetic layer divided by the nonmagnetic layer for achieving noise reduction.

[0043]

The underlying layer of the magnetic layer may be selected depending on the magnetic layer. Examples of the underlying layer include an underlying layer composed of at least one or more materials selected from non-magnetic metals such as Cr, Mo, Ta, Ti, W, V, B and Al, or oxides, 30 nitride, carbides and the like of those metals. For a magnetic layer comprising Co as the main component, an underlying layer of pure Cr or Cr alloy is preferred in view of improving magnetic characteristics. The underlying layer is not limited to a monolayer, and may 35 be a plural-layered structure in which identical or

different layers are laminated. For example, a multi-layer underlying layer such as Al/Cr/CrMo and Al/Cr/Cr is exemplified.

The unevenness control layer for preventing absorption of magnetic disk to magnetic head may be provided between the substrate and the magnetic layer or on the magnetic layer. Because surface roughness of the disk is properly controlled by providing the unevenness control layer, the magnetic disk is prevented from being absorbed to the magnetic head and hence a highly reliable magnetic disk can be provided. Various materials and production methods for the unevenness control layer have been known and they are not particularly limited. For example, the material of the unevenness control layer may be one or more metals selected from Al, Ag, Ti, Nb, Ta, Bi, Si, Zr, Cr, Cu, Au, Sn, Pd, Sb, Ge, Mg and the like, alloys thereof, oxides, nitrides, carbides thereof and the like. For the ease of production, metals containing Al as a main component such as pure Al, Al alloys, Al oxides and Al nitrides are preferred.

[0044]

In addition, taking head stiction into consideration, surface roughness of the unevenness forming layer is preferably  $R_{max}$  of 50-300 Å. More preferable range is  $R_{max}$  of 100-200 Å. When the  $R_{max}$  is less than 50 Å, the disk surface is nearly flat, and hence the magnetic head and the magnetic disk are absorbed to each other. This may disadvantageously cause damage of the magnetic head and the magnetic disk, and head crash due to adsorption. In addition, when the  $R_{max}$  exceeds 300 Å, glide height becomes larger and recording density is disadvantageously lowered.

It is to be noted that unevenness may be provided on the surface of the glass substrate by means such as etching treatment and irradiation of laser lights without providing the unevenness control layer, and subjecting to a texturing

treatment.

[0045]

The protective layer may be, for example, Cr film, Cr alloy film, carbon film, zirconia film, silica film or the like. These protective films can be successively formed by an inline-type sputtering apparatus and the like together with the underlying layer, the magnetic layer and the like. These protective films may be either monolayer or may have multilayer structure composed of identical or different layers.

Another protective layer may be provided on or instead of the protective layer explained above. For example, a silicon oxide ( $\text{SiO}_2$ ) film may be formed on the protective layer mentioned above by applying tetraalkoxysilane diluted in an alcoholic solvent, in which colloidal silica microparticles are dispersed, and applying and further sintering. In this case, it functions both as a protective layer and as an unevenness control layer.

While various kinds of layers have been proposed as the lubricating layer, it is generally formed by applying a liquid lubricating agent, perfluoropolyether, diluted in a solvent such as freons by dipping, spin coating, spraying or the like and subjecting to a heat-treatment as required.

25 [0046]

[Examples]

The details of the present invention will be explained in the following examples, however, the present invention is not limited to these examples.

30 In Tables 1, the glass compositions of Examples 1 to 15 are listed with respect to mol%. As the starting materials during melting these glasses,  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{Al}(\text{OH})_3$ ,  $\text{MgO}$ ,  $\text{Y}_2\text{O}_3$ ,  $\text{TiO}_2$ ,  $\text{ZrO}_2$ ,  $\text{KNO}_3$ ,  $\text{Sr}(\text{NO}_3)_2$  and the like were weighed into 250-300 g portions according to the given compositions 35 shown in Tables 1 and 2 and mixed sufficiently to provide

formulated batches. It was charged in a platinum crucible and melted in air for 4 to 5 hours at 1550°C with stirring to melt a glass. After melting, the glass melting liquid was cast into a carbon metal mold having a size of 180  
5  $\times$  15  $\times$  25 mm, left to cool to the glass transition temperature, immediately transferred into an annealing furnace, annealed in the glass transition temperature range for about 1 hour and left to cool to room temperature in the furnace. In  
10 the resulting glasses, crystals which can be observed by a microscope did not deposit.

[0047]

After the glass having a size of 180  $\times$  15  $\times$  25 mm were polished to a size of 100  $\times$  10  $\times$  10 mm, 10  $\times$  10  $\times$  20 mm or 10  
15  $\times$  1  $\times$  20 mm, they were put in a heat treatment furnace, elevating a temperature at an elevating speed of temperature ranging from 1 to 5 °C/min up to the first heat treatment temperature described in Tables 1 and 2, carrying out the first heat treatment while keeping the temperature for about 2 to 10 hours, and immediately after finishing the first  
20 heat treatment, elevating a temperature at an elevating speed of temperature ranging from 2 to 10 °C/min from the first heat treatment temperature to the second heat treatment temperature described in Tables 1 and 2. After keeping the temperature for about 1 to 5 hours, the  
25 crystallized glass was produced by cooling to the room temperature in the furnace. The crystallized glass thus obtained was further polished as to have the length of 95 mm, and then used as a sample for measuring the Young's modulus and the specific gravity. Measurements of the  
30 Young's modulus were carried out using a 95  $\times$  10  $\times$  10 mm sample by ultrasonic method. The data obtained by the measurements are listed together with the glass compositions in Tables 1 and 2.

For comparison, the ion-exchanged glass substrate  
35 disclosed in Japanese Patent Unexamined Publication

No. Hei. 1-239036 and the glass substrate disclosed in U.S. Patent No. 2,516,553 were utilized as Comparative examples 1 and 2 and their compositions and characteristics are shown in Table 3.

[ 0 0 4 8 ]

[ T a b l e 1 ]

C o m p o s i t i o n s o f c r y s t a l l i z e d g l a s s e s o f e x a m p l e s ( m o l % )

Composition	1	2	3	4	5	6	7	8	9	10
SiO <sub>2</sub>	48.00	47.00	47.00	46.00	47.00	41.00	43.00	45.00	47.00	49.00
Al <sub>2</sub> O <sub>3</sub>	11.00	10.50	10.50	10.50	12.50	12.50	12.50	12.50	12.50	10.50
MgO	30.00	30.00	28.50	31.00	28.50	34.50	32.50	30.50	28.50	29.50
ZnO										
NiO										
Y <sub>2</sub> O <sub>3</sub>	1.00	0.50		0.50	2.00	2.00	2.00	2.00	2.00	1.00
ZrO <sub>2</sub>	0.00	2.00	2.00	2.00		0.00	0.00	0.00	0.00	
TiO <sub>2</sub>	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00
Al <sub>2</sub> O <sub>3</sub> /MgO	0.37	0.35	0.37	0.34	0.44	0.36	0.38	0.41	0.44	0.35
Transition temp. T <sub>g</sub> (°C)	732	735	729	732	729	738	740	739	740	732
Pretreatment temp.(°C)	T <sub>g</sub> +28	T <sub>g</sub> +35	T <sub>g</sub> +31	T <sub>g</sub> +38	T <sub>g</sub> +31	T <sub>g</sub> +22	T <sub>g</sub> +30	T <sub>g</sub> +31	T <sub>g</sub> +20	T <sub>g</sub> +28
Pretreatment time(h)	4	4	4	4	4	4	4	4	4	4
Elevating speed of temp.(°C/h)	300	300	300	300	300	300	300	300	300	300
Second treatment temp.(°C)	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
Second treatment time(h)	4	4	4	4	4	4	4	4	4	4
Elevating speed of temp.(h)	240	240	240	240	240	240	240	240	240	240
Kind of fracture surface	Glass									
Transparency at wavelength of 600nm	75%	73%	78%	79%	76%	75%	73%	78%	79%	76%
Specific gravity(g/cm <sup>3</sup> )	3.086	3.14	3.11	3.158	3.038	3.309	3.254	3.207	3.168	3.068
Young's modulus(GPa)	148.5	150.5	147	153.2	146.7	179.1	170	163.4	157.3	149.1
Poisson ratio	0.231	0.23	0.229	0.23	0.225	0.245	0.245	0.241	0.237	0.228
Kind of main crystals	Enstatite									
Kind of other crystals	Titanate									
Specific modulus of elasticity MNm/kg	48.1	47.9	47.3	48.5	48.3	54.1	52.2	51.0	49.7	48.6
Thermal expansion coefficient 10 <sup>-7</sup> /°C	72	72	75	74	70	83	81	76	72	77
Average particle diameter(nm)	30·40	30·40	40·50	20·30	50·70	100·150	80·120	50·70	50·70	40·50

Enstatite: enstatite and its solid solution; Titanate: salt of titanic acid

[ 0 0 4 9 ]

[ Table 2 ]

Compositions of crystallized glasses of examples (mol%)

Composition	11	12	13	14	15
SiO <sub>2</sub>	46.00	46.00	46.00	46.00	46.00
Al <sub>2</sub> O <sub>3</sub>	10.50	10.50	10.50	10.50	10.50
MgO	30.50	30.00	30.00	31.00	30.00
K <sub>2</sub> O	0.50				0.50
SrO		1.00	1.50	1.00	0.50
Y <sub>2</sub> O <sub>3</sub>	0.50	0.50		0.50	0.50
ZrO <sub>2</sub>	2.00	2.00	2.00	2.00	2.00
TiO <sub>2</sub>	10.00	10.00	9.00	9.00	10.00
Al <sub>2</sub> O <sub>3</sub> /MgO	0.34	0.35	0.35	0.34	0.34
Transition temp. Tg (°C)	726	728	726	725	727
Pretreatment temp.(°C)	Tg+30	Tg+30	Tg+30	Tg+30	Tg+30
Pretreatment time(h)	4	4	4	4	4
Elevating speed of temp.(°C/h)	300	300	300	300	300
Second treatment temp.(°C)	1000	1000	1000	1000	1000
Second treatment time(h)	4	4	4	4	4
Elevating speed of temp.(°C/h)	240	240	240	240	240
Kind of fracture surface	Glass	Glass	Glass	Glass	Glass
Transparency at wavelength of 600nm	82%	70%	67%	80%	80%
Specific gravity(g/cm <sup>3</sup> )	3.127	3.172	3.175	3.124	3.15
Young's modulus (GPa)	149.2	151.8	152.1	147.1	148.2
Poisson ratio	0.232	0.234	0.234	0.231	0.232
Kind of main crystals	Enstatite	Enstatite	Enstatite	Enstatite	Enstatite
Kind of other crystals	Titanate	Titanate	Titanate	Titanate	Titanate
Specific modulus of elasticity(MNm/kg)	47.7	47.9	47.9	47.1	47.0
Thermal expansion coefficient(10 <sup>-7</sup> /°C)	77.9	75.1	74.7	79.2	78.1
Average particle diameter(nm)	20-30	30-50	30-50	20-30	20-30

[0050]

[Table 3]

Comparative Example	1	2
	Chemically reinforced glass	Commercially available TS-10 crystallized glass
Oxide	Japanese patent Unexamined Publication No.Hei.1-239036	U.S.Patent No. 2516553
SiO <sub>2</sub>	73.0	
Al <sub>2</sub> O <sub>3</sub>	0.6	
CaO	7.0	
Na <sub>2</sub> O	9.0	
K <sub>2</sub> O	9.0	
ZnO	2.0	
As <sub>2</sub> O <sub>3</sub>	0.2	
Young's modulus(GPa)	79	90-100
Surface roughness Ra (nm)	12	10-35

5 [0051]

As seen from the results shown in Tables 1 and 2, the crystallized glasses of the present invention in Examples 1 to 15 had characteristics of strength, such as the Young's modulus (equal to or higher than 140 GPa), the specific modulus of elasticity (in the range of 40 to 60 MNm/kg) and the like in a large value. Therefore, in the case of using these glasses as substrates for information recording medium such as a magnetic recording medium, even if these glass substrates are rotated at a high speed, they are not likely to exhibit warp or walking, and hence it is understood

that they can meet the demand of further thinner substrates. In addition, when the liquid phase temperature was measured with respect to glasses before heat treatment in Example 1, Example 4 and Example 10, the temperature was 1300 °C, 5 1290 °C and 1270 °C, respectively, which satisfy the liquid phase temperature (for example, equal to or less than 1350 °C) required from the viewpoint of glass melting and molding. In addition, when the average particle diameter of the crystal particle was measured with a Transmission Electron 10 Microscope (TEM) with respect to the crystallized glass in Examples 1 to 15, the particles having the average particle diameter ranging from 20-30nm to 100-150nm were observed. In addition, the polished planes subjected to optical glass polishing for surface roughness measurement 15 were subjected to surface observation with an Atomic Force Microscope (AFM) with respect to the crystallized glass in Examples 1-4 and 10. From the results, the surface roughness ( $R_a$ ) was equal to or less than 5 Å. The surface roughness ( $R_a$ ) of these crystallized glasses can be polished to equal to or less than 5 Å with, for example, the polishing method for conventional optical glass using abrasives such as synthesis diamond, silicon carbide, calcium oxide, iron oxide, cerium oxide. Therefore, substrates excellent in flatness can be obtained and it is useful as glass substrates 20 for magnetic recording media for the purpose of smaller flying height. The crystallized glass of the present invention had the light transparency equal to or higher than 50 % at a wavelength of 600nm in the case of the thickness of 1 mm and had transparency to some extent. Such 25 transparency can be an index showing whether desirable kinds of crystals and particle diameter of crystals are obtained. In the case of the crystallized glass of the present invention, said transparency can range, for example, from 30 60 to 90 %.

35 [0052]

On the contrary, while the chemically reinforced glass substrate in Comparative Example 1 shown in Table 3 was excellent in the surface smoothness and flatness, it was much inferior in characteristics of strength such as heat resistance and the Young's modulus than the crystallized glass of the present invention. Accordingly, when magnetic recording media were produced, heat treatment to a magnetic layer for obtaining high coercive force could not be carried out sufficiently, thereby the magnetic recording media having high coercive force could not be obtained. In addition, the glass in Comparative Example 1 contained large amount of alkali, so that the collosion of the magnetic film with the substrate easily occurred, thereby it was afraid that the magnetic film was damaged.

In addition, the crystallized glass substrate in Comparative Example 2 was inferior in the Young's modulus and smoothness than the glass of the present invention. In particular, because the smoothness of the substrate was deteriorated by the existence of large crystal particles, it was difficult to attempt high-density recording.

[0053]

[Effects of the Invention]

The crystallized glass substrate for information recording medium of the present invention can be easily molded, and has the large Young's modulus equal to or higher than 140 GPa, high heat resistance, excellent surface productivity and surface smoothness (less than 10 Å of surface roughness Ra), as well as it can be used as a substrate material having large hardness and strength.

In addition, because the materials of the substrate composed of the crystallized glass of the present invention is excellent in heat resistance, heat treatment required for improving characteristics of magnetic films can be used without deformation of substrates. Because it is excellent in flatness, smaller flying height of magnetic head, that

is, high-density recording, can be accomplished. Because it has high Young's modulus, specific modulus of elasticity and strength, it has advantages that thinner magnetic disks can be obtained and the magnetic disks can rotate at a high speed while avoiding the fracture of the magnetic disks.

In addition, the crystallized glass of the present invention can be produced with relative stability, and production in the industrial scale can be done easily, so that it can be greatly expected as substrate glasses for cheap magnetic recording media in the next generation.

[Document] Abstract

[Abstract]

[Problems] To provide a substrate for information recording medium composed of such a crystallized glass as having high  
5 Young's modulus, strength and heat resistance, being excellent in surface smoothness, surface homogeneity and surface processability, as well as having a relatively low temperature of glass liquid phase and being capable of producing cheaply, and an information recording medium  
10 using this substrate.

[Means for solving problems] A crystallized glass substrate for information recording medium comprising 35-65 mol% of SiO<sub>2</sub>, 5-25 mol% of Al<sub>2</sub>O<sub>3</sub>, 10-40 mol% of MgO and 5-15 mol% of TiO<sub>2</sub>, wherein the sum of the above components  
15 is at least equal to or more than 93mol%, and having a composition wherein a molar ratio (Al<sub>2</sub>O<sub>3</sub>/MgO) is less than 0.5, and wherein a main crystal phase is enstatite and/or its solid solution. An information recording medium having the substrate and a recording layer formed on the substrate.  
20 [Selected figure]